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N-Nitro-1*H*-pyrrole-2-carboxamide

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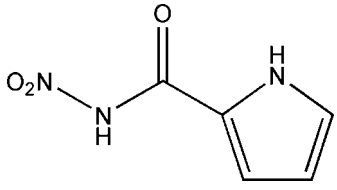
Key indicators: single-crystal X-ray study; T = 133 K; mean σ(C–C) = 0.002 Å; R factor = 0.043; wR factor = 0.106; data-to-parameter ratio = 13.0.

In the title compound, $C_5H_5N_3O_3$, the nitro group is twisted with respect to the amide group, with C—N—N—O torsion angles of 29.0 (2) and −153.66 (14)°. In the crystal, molecules are linked through intermolecular N—H···O and C—H···O hydrogen bonds, forming supramolecular chains along the *a* axis. These chains stack in parallel and form distinct layer motifs in the (001) plane.

Related literature

For applications of pyrrole derivatives as antimicrobials, see: Mohamed *et al.* (2009). For the structures of similar pyrrole derivatives, see: Zeng *et al.* (2007, 2010); Wang *et al.* (2010); Ferreira *et al.* (2002). For the synthesis of *N*,*N*′-dinitrourea (DNU), see: Goede *et al.* (2001).



Experimental

Crystal data

$C_5H_5N_3O_3$
M_r = 155.12
Orthorhombic, *Pbca*
a = 9.988 (3) Å
b = 6.4547 (17) Å
c = 19.184 (6) Å
V = 1236.8 (6) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 133 K
0.47 × 0.43 × 0.20 mm

Data collection

Rigaku AFC10/Saturn724+ diffractometer
8849 measured reflections
1402 independent reflections
1214 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.106
S = 1.00
1402 reflections
108 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···O3^{i}	0.88 (3)	2.21 (3)	3.001 (2)	150 (2)
N2—H2*N*···O1^{ii}	0.88 (2)	2.11 (2)	2.982 (2)	171.5 (19)
C3—H3···O1^{ii}	0.95	2.39	3.269 (2)	154
C3—H3···O2^{ii}	0.95	2.48	3.245 (2)	138

Symmetry codes: (i) $x-\frac{1}{2}, -y+\frac{1}{2}, -z+1$; (ii) $x+\frac{1}{2}, -y+\frac{1}{2}, -z+1$.

Data collection: *CrystalClear* (Rigaku, 2008); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2382).

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supplementary materials

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N-Nitro-1*H*-pyrrole-2-carboxamide

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Comment

Pyrrole derivatives play an important role in heterocyclic chemistry due to their intrinsic biological activities as antimicrobial agents (Mohamed *et al.*, 2009). The structures of these compounds have been reported extensively, such as 2,3,5-substituted pyrrole derivatives (Ferreira *et al.*, 2002), 1-Benzyl-*N*-methyl-1*H*-pyrrole-2-carboxamide (Zeng *et al.*, 2010), 2-(4,5-dibromo-1*H*-pyrrole-2-carboxamido) propionate (Zeng *et al.*, 2007) and Tetraethyl 1,1'-(ethane-1,2-diyl)bis(2,5- dimethyl-1*H*-pyrrole-3,4-dicarboxylate) (Wang *et al.*, 2010).

The bond length of N2—C5 for the title compound (1.404 (2) Å) is about 0.07 Å longer than compound 1-Benzyl-*N*-methyl-1*H*-pyrrole-2-carboxamide (1.334 (3) Å) (Zeng *et al.*, 2010) (Fig. 1). The unit is nearly co-planar with the twist happens at nitro group (C5—N2—N3—O2 = 29.0 (2), C5—N2—N3—O3 = -153.66 (14)), the maximum deviation of other torsions is C3—C4—C5—N2 = -8.6 (3)°.

In the crystal structure (Fig. 2), molecules are connected through N1—H1N···O3, N2—H2N···O1, C3—H3···O1, C3—H3···O3 (Table 1) hydrogen bonds to form one-dimensional supramolecular chains along the *a* axis. These supramolecular chains stack in parallel and form distinct layer motif in (0 0 1) plane.

Experimental

Pyrrole (0.67 g, 0.01 mol) was added to a solution of *N*,*N*'-dinitrourea (DNU) (1.5 g, 0.01 mol) dissolved in acetonitrile (10 ml), stirred at room temperature for 24 h, the crude compound was obtained after acetonitrile was evaporated. Then the products were dissoved in ethyl acetate, colourless crystals suitable for X-ray crystal diffraction were obtained by slow evaporation of the solution at room temperature. DNU was synthesized according to the literautre (Goede *et al.*, 2001).

Refinement

The hydrogen atoms bonded to N1 and N2 were located from a difference Fourier maps and refined isotropically with N—H = 0.88 (3) Å and 0.88 (2) Å respectively. The remaining hydrogen atoms were geometrically positioned (all C—H = 0.9500 Å).

Figures

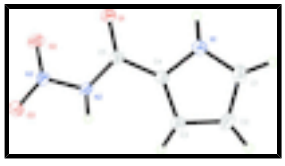

Fig. 1. Thermal ellipsoid plot of $C_5H_5N_3O_3$ at the 50% probability level; Hydrogen atoms are drawn as spheres of arbitrary radius.

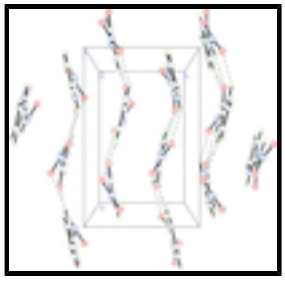

Fig. 2. Hydrogen-bonded layer structure.

N-Nitro-1*H*-pyrrole-2-carboxamide

Crystal data

$C_5H_5N_3O_3$	$F(000) = 640$
$M_r = 155.12$	$D_x = 1.666$ Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation, λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 3166 reflections
$a = 9.988$ (3) Å	θ = 3.2–27.5°
$b = 6.4547$ (17) Å	μ = 0.14 mm^{-1}
$c = 19.184$ (6) Å	$T = 133$ K
$V = 1236.8$ (6) Å^3	Platelet, colourless
$Z = 8$	0.47 × 0.43 × 0.20 mm

Data collection

Rigaku AFC10/Saturn724+ diffractometer	1214 reflections with $I > 2\sigma(I)$
Radiation source: Rotating Anode	$R_{int} = 0.034$
graphite	$\theta_{max} = 27.5°$, $\theta_{min} = 3.9°$
Detector resolution: 28.5714 pixels mm^{-1}	$h = -12 \rightarrow 12$
φ and ω scans	$k = -8 \rightarrow 8$
8849 measured reflections	$l = -24 \rightarrow 24$
1402 independent reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.043$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.106$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.00$	$w = 1/[\sigma^2(F_o^2) + (0.0596P)^2 + 0.536P]$ where $P = (F_o^2 + 2F_c^2)/3$
1402 reflections	$(\Delta/\sigma)_{max} < 0.001$
108 parameters	$\Delta\rho_{max} = 0.34$ e Å^{-3}

0 restraints $\Delta\rho_{min} = -0.15$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.17512 (12)	0.34028 (18)	0.50500 (5)	0.0219 (3)
O2	0.27334 (11)	0.3909 (2)	0.37665 (6)	0.0256 (3)
O3	0.45611 (12)	0.21230 (19)	0.36431 (6)	0.0255 (3)
N1	0.25340 (14)	0.3489 (2)	0.64493 (7)	0.0207 (3)
N2	0.39562 (13)	0.3045 (2)	0.47037 (7)	0.0182 (3)
N3	0.37143 (13)	0.3012 (2)	0.39910 (7)	0.0178 (3)
C1	0.31849 (18)	0.3310 (3)	0.70652 (8)	0.0235 (4)
H1	0.2800	0.3496	0.7514	0.028*
C2	0.44956 (17)	0.2815 (3)	0.69312 (8)	0.0240 (4)
H2	0.5173	0.2581	0.7270	0.029*
C3	0.46594 (16)	0.2715 (2)	0.62056 (8)	0.0198 (4)
H3	0.5464	0.2417	0.5962	0.024*
C4	0.34176 (15)	0.3137 (2)	0.59132 (8)	0.0160 (3)
C5	0.29335 (15)	0.3204 (2)	0.52027 (8)	0.0158 (3)
H1N	0.169 (3)	0.380 (4)	0.6375 (12)	0.049 (7)*
H2N	0.474 (2)	0.250 (3)	0.4794 (11)	0.032 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0140 (6)	0.0339 (7)	0.0177 (5)	−0.0002 (5)	−0.0004 (4)	0.0003 (5)
O2	0.0181 (6)	0.0398 (7)	0.0188 (6)	0.0049 (5)	−0.0022 (4)	0.0071 (5)
O3	0.0198 (6)	0.0381 (7)	0.0187 (6)	0.0050 (5)	0.0027 (5)	−0.0052 (5)
N1	0.0164 (7)	0.0276 (7)	0.0180 (6)	0.0021 (6)	0.0007 (5)	0.0002 (6)
N2	0.0132 (6)	0.0281 (7)	0.0133 (6)	0.0016 (5)	−0.0022 (5)	0.0011 (5)
N3	0.0153 (6)	0.0241 (7)	0.0141 (6)	−0.0022 (5)	0.0004 (5)	0.0013 (5)
C1	0.0273 (9)	0.0286 (9)	0.0145 (7)	−0.0002 (7)	0.0011 (6)	0.0000 (6)
C2	0.0223 (8)	0.0315 (9)	0.0183 (8)	−0.0013 (7)	−0.0053 (6)	0.0015 (6)
C3	0.0146 (7)	0.0257 (8)	0.0191 (8)	−0.0008 (6)	−0.0001 (6)	0.0010 (6)
C4	0.0147 (7)	0.0176 (7)	0.0158 (7)	−0.0013 (6)	0.0003 (6)	−0.0001 (5)
C5	0.0148 (7)	0.0164 (7)	0.0163 (7)	−0.0011 (6)	−0.0002 (5)	−0.0002 (6)

Geometric parameters (Å, °)

O1—C5	1.2234 (19)	N2—H2N	0.88 (2)
O2—N3	1.2167 (17)	C1—C2	1.372 (2)
O3—N3	1.2206 (17)	C1—H1	0.9500
N1—C1	1.354 (2)	C2—C3	1.403 (2)
N1—C4	1.374 (2)	C2—H2	0.9500
N1—H1N	0.88 (3)	C3—C4	1.388 (2)
N2—N3	1.3886 (18)	C3—H3	0.9500
N2—C5	1.404 (2)	C4—C5	1.447 (2)
C1—N1—C4	109.32 (14)	C1—C2—C3	107.93 (15)
C1—N1—H1N	128.4 (16)	C1—C2—H2	126.0
C4—N1—H1N	122.3 (16)	C3—C2—H2	126.0
N3—N2—C5	123.09 (13)	C4—C3—C2	106.72 (14)
N3—N2—H2N	110.2 (14)	C4—C3—H3	126.6
C5—N2—H2N	123.1 (14)	C2—C3—H3	126.6
O2—N3—O3	126.02 (14)	N1—C4—C3	107.68 (13)
O2—N3—N2	118.77 (13)	N1—C4—C5	119.04 (14)
O3—N3—N2	115.15 (13)	C3—C4—C5	133.25 (14)
N1—C1—C2	108.33 (14)	O1—C5—N2	123.14 (14)
N1—C1—H1	125.8	O1—C5—C4	123.46 (14)
C2—C1—H1	125.8	N2—C5—C4	113.39 (13)
C5—N2—N3—O2	29.0 (2)	C2—C3—C4—C5	−177.88 (16)
C5—N2—N3—O3	−153.66 (14)	N3—N2—C5—O1	−2.7 (2)
C4—N1—C1—C2	−0.71 (19)	N3—N2—C5—C4	177.98 (13)
N1—C1—C2—C3	0.9 (2)	N1—C4—C5—O1	−5.8 (2)
C1—C2—C3—C4	−0.67 (19)	C3—C4—C5—O1	172.13 (17)
C1—N1—C4—C3	0.28 (18)	N1—C4—C5—N2	173.46 (13)
C1—N1—C4—C5	178.72 (14)	C3—C4—C5—N2	−8.6 (3)
C2—C3—C4—N1	0.24 (18)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N···O3^{i}	0.88 (3)	2.21 (3)	3.001 (2)	150 (2)
N2—H2N···O1^{ii}	0.88 (2)	2.11 (2)	2.982 (2)	171.5 (19)
C3—H3···O1^{ii}	0.95	2.39	3.269 (2)	154
C3—H3···O2^{ii}	0.95	2.48	3.245 (2)	138

Symmetry codes: (i) *x*−1/2, −*y*+1/2, −*z*+1; (ii) *x*+1/2, −*y*+1/2, −*z*+1.

Fig. 1

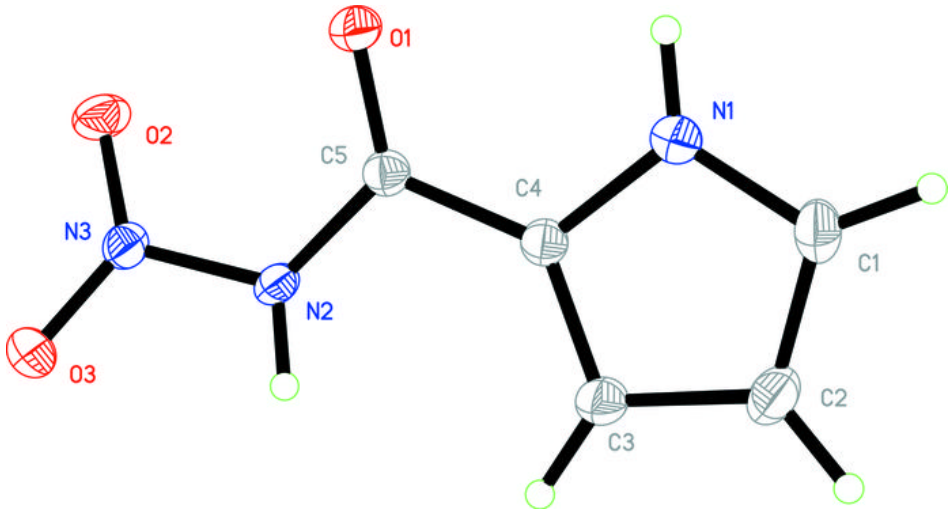

Fig. 2